University of Groningen

# Synthesis of new bis(cyclopentadienyl)yttrium complexes with ether functionalized cyclopentadienyl ligands. Crystal structure of [(C5H4CH2CH2OMe)2Y(μ-H)2BH2]

Laske, Dietmar A.; Duchateau, Robbert; Teuben, Jan H.; Spek, Anthony L.

*Published in:*
Journal of Organometallic Chemistry

*DOI:*
10.1016/0022-328X(93)83352-V

**IMPORTANT NOTE: You are advised to consult the publisher's version (publisher's PDF) if you wish to cite from it. Please check the document version below.**

*Document Version*
Publisher's PDF, also known as Version of record

*Publication date:*
1993

Link to publication in University of Groningen/UMCG research database

*Citation for published version (APA):*
Laske, D. A., Duchateau, R., Teuben, J. H., & Spek, A. L. (1993). Synthesis of new bis(cyclopentadienyl)yttrium complexes with ether functionalized cyclopentadienyl ligands. Crystal structure of [(C5H4CH2CH2OMe)2Y(μ-H)2BH2]. *Journal of Organometallic Chemistry*, *462*(1). https://doi.org/10.1016/0022-328X(93)83352-V

**Copyright**
Other than for strictly personal use, it is not permitted to download or to forward/distribute the text or part of it without the consent of the author(s) and/or copyright holder(s), unless the work is under an open content license (like Creative Commons).

The publication may also be distributed here under the terms of Article 25fa of the Dutch Copyright Act, indicated by the "Taverne" license. More information can be found on the University of Groningen website: https://www.rug.nl/library/open-access/self-archiving-pure/taverne-amendment.

**Take-down policy**
If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

*Downloaded from the University of Groningen/UMCG research database (Pure): http://www.rug.nl/research/portal. For technical reasons the number of authors shown on this cover page is limited to 10 maximum.*

JOM 23716

# Synthesis of new bis(cyclopentadienyl)yttrium complexes with ether functionalized cyclopentadienyl ligands. Crystal structure of $[(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}H)_2BH_2]$ *

Dietmar A. Laske, Robbert Duchateau, Jan H. Teuben

*Groningen Center for Catalysis and Synthesis, Department of Chemistry, University of Groningen, Nijenborgh 4, 9747 AG Groningen (Netherlands)*

Anthony L. Spek

*Bijvoet Center for Biomolecular Research, Laboratory of Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht (Netherlands)*

(Received March 17, 1993)

**Abstract**

Treatment of $C_5H_4CH_2CH_2OMe \cdot Li(TMEDA)$ (TMEDA = N, N, N´, N´ tetramethylethylenediamine) with half an equivalent of $YCl_3(THF)_{3.5}$ in toluene afforded the salt- and solvent-free dimeric biscyclopentadienyl yttrium chloride $[(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}Cl)]_2$ (**1**). Reaction of **1** with one equivalent of $LiAlH_4$ in THF gave the yttrium alumohydride complex $[(C_5H_4CH_2CH_2OMe)_2YAlH_4]$ (**2**), which was transformed into $[(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}H)]_2$ (**4**) by expulsion of $AlH_3$ with $NEt_3$. Reaction of **1** with an excess of $LiBH_4$ gave the yttrium borohydride complex $[(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}H)_2BH_2]$ (**3**), whose crystal structure has been shown to be monomeric species with yttrium-coordinated ether functions and a $\eta^2$-bonded borohydride ligand.

## 1. Introduction

Salt and solvent free hydrides of Group 3 metals and the lanthanides $[Cp´_2Ln(\mu\text{-}H)]_2$ ($Cp´ = C_5H_{5-n}R_n$, R = alkyl) are extremely interesting because of their potential as catalysts for olefin polymerization, hydrogenation, hydrosilylation, and hydroamination, and have therefore been widely investigated [1-4]. A frequently used method to synthesize these hydrides involves hydrogenolysis of the corresponding carbyls $Cp´_2LnR$, but this route is cumbersome [5]. A more attractive, direct, route is through reaction of $Cp´_2LnCl$ and $LiAlH_4$ to give $Cp´_2LnAlH_4$, which on treatment with $Et_3N$ produces $[Cp´_2Ln(\mu\text{-}H)]_2$ [6].

The compounds $Cp´_2LnX$ (X = halide, hydride, carbyl) are normally so strongly Lewis acidic that they tend to incorporate salt (*e.g.* LiCl or $MgCl_2$) or solvent molecules. These are generally very difficult to remove, and greatly reduce the (catalytic) reactivity of the metal centre. On the other hand a Lewis base function on each of the spectator ligands can prevent inclusion of salt and solvent and enhance the activity and selectivity of the metal centre. We therefore planned a series of dicyclopentadienyl derivatives of yttrium, in which the cyclopentadienyl ligand carries a pending ether function, and chose the system $C_5H_5CH_2CH_2OMe$. During our investigations analogous derivatives were reported for La, Pr and Nd [7], and this has prompted us to present our results on yttrium.

## 2. Results and discussion

Convenient amounts (50 mmol scale) of the starting material $[(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}Cl)]_2$ (**1**) were obtained by treatment of a solution of $C_5H_4CH_2CH_2OMe \cdot Li(TMEDA)$ in toluene at -80°C with half an equivalent of $YCl_3(THF)_{3.5}$ followed by overnight stirring at room temperature. Filtration, concentration

Correspondence to: Professor J.H. Teuben.

* This paper is dedicated to Professor M.F. Lappert F.R.S. on the occasion of his 65th birthday.

0022-328X/93/$6.00

© 1993 - Elsevier Sequoia S.A. All rights reserved

and cooling of the solution gave white crystals of **1** (eqn. (1)).

$$2C_5H_4CH_2CH_2OMe \cdot Li(TMEDA) + YCl_3(THF)_{3.5} \longrightarrow \underset{\mathbf{1}}{\tfrac{1}{2}[(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}Cl)]_2} \quad (1)$$

Reaction of **1** with one equivalent of $LiAlH_4$ gave the yttrium alumohydride complex $(C_5H_4CH_2CH_2OMe)_2YAlH_4$ (**2**) as white crystals (eqn. (2)). The borohydride derivative **3** was prepared analogously (eqn. (2)).

$$\underset{\mathbf{1}}{\tfrac{1}{2}[(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}Cl)]_2} \xrightarrow[\text{-LiCl}]{LiMH_4} (C_5H_4CH_2CH_2OMe)_2YMH_4 \quad (2)$$

**2** M = Al;
**3** M = B

$$(C_5H_4CH_2CH_2OMe)_2YAlH_4 \xrightarrow[NEt_3]{50^\circ C} \underset{\mathbf{2}}{} \tfrac{1}{2}[\underset{\mathbf{4}}{(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}H)}]_2 + AlH_3 \cdot NEt_3 \quad (3)$$

Treatment of **2** with an excess of $NEt_3$ in a mixture of toluene and pentane (1:3) led to expulsion of $AlH_3$ [8*] and formation of the symmetric yttrium hydride dimer $[(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}H)]_2$ (**4**) in moderate yield (eqn. (3)) [9*]. Direct conversion of **1** into **4** can be easily carried out by *in situ* reaction and this presents an excellent route for the synthesis of hydride **4**. No attempt to obtain monometallic hydrides by the well-known treatment of borohydrides with triethylamine (*e.g.* for $Cp_2Zr(BH_4)_2$ this procedure yields triethylaminoborane and zirconium monohydride or zirconium dihydride [10*]) succeeded in displacing $BH_3$ from **3** to give **4** (similar reactions of samarocene borohydrides $Cp'_2SmBH_4$ ($Cp' = C_5H_5$, $C_5H_4{}^tBu$, $C_5H_3{}^tBu_2$) with triethylamine were also unsuccessful [6b]).

The crystal structure of **3** was determined (Fig. 1). The molecular structure is disordered in that for C17 there are two possible positions, each with *ca.* 50% occupancy (for the sake of clearness one position is shown in Fig. 1). The bond parameters (see Table 1) indicate that the molecule has a pseudo-octahedral coordination geometry around yttrium if the $\eta^5$-bonded cyclopentadienyl ligand is regarded as occupying a single polyhedral vertex. The yttrium atom appears to be electronically saturated, since all the ligand contributions, when accounted for in the regular way, add up to 18 electrons on the metal. The Y-Cent1 (2.386(3) Å) and Y-Cent2 (2.393(5) Å) distances are similar to those in $[(MeC_5H_4)_2Y(\mu\text{-}H)THF]_2$ (2.397, 2.414 Å) [11]. Two hydride atoms bridge between yttrium (Y-H3 = 2.26(5), Y-H4 = 2.27(5) Å) and boron, and the latter also bears two terminal hydrides. The borohydride acts as a bidentate ligand, which is common for analogous compounds in which the metal is smaller than or comparable with yttrium (Ti, Yb), whereas for

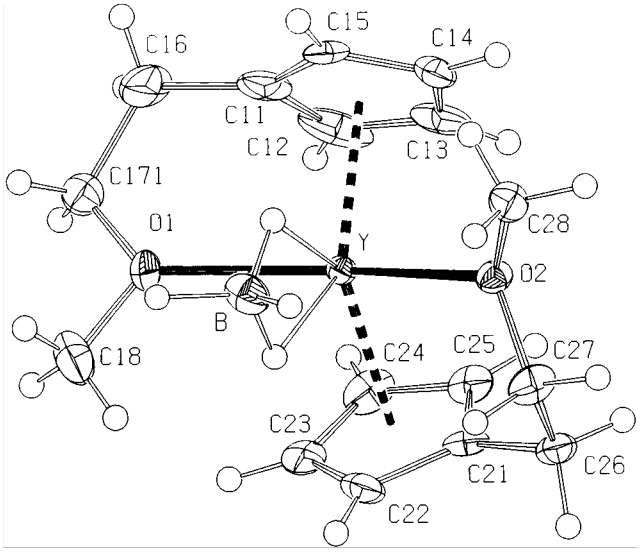


Fig. 1. Atomic displacement ellipsoid plot (50% probability level) of $(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}H)_2BH_2$ (**3**).

TABLE 1. Selected bond distances (Å) and angles (°) for $(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}H)_2BH_2$ (**3**)

| | | | |
|---|---|---|---|
| *Bond distances* | | | |
| Y-O1 | 2.438(5) | Y-O2 | 2.419(4) |
| Y-C11 | 2.659(8) | Y-C21 | 2.649(6) |
| Y-C12 | 2.639(8) | Y-C22 | 2.694(10) |
| Y-C13 | 2.668(6) | Y-C23 | 2.716(10) |
| Y-C14 | 2.695(9) | Y-C24 | 2.671(1) |
| Y-C15 | 2.678(6) | Y-C25 | 2.631(10) |
| Y-H3 | 2.26(5) | Y-H4 | 2.27(5) |
| B-H1 | 1.16(5) | B-H2 | 1.15(6) |
| B-H3 | 1.11(5) | B-H4 | 1.11(5) |
| C16-C171 | 1.54(2) | C16-C172 | 1.27(3) |
| O1-C171 | 1.394(14) | O1-C172 | 1.53(2) |
| Y-Cent1 [a] | 2.386(3) | Y-Cent2 [a] | 2.393(5) |
| *Bond angles* | | | |
| H3-Y-H4 | 43.8(18) | H3-B-H4 | 99 (4) |
| Y-H4-B | 108 (3) | H1-B-H2 | 103 (4) |
| Y-H3-B | 109 (3) | O2-C27-C26 | 108.6(5) |
| Y-O1-C171 | 128.3(6) | Y-O1-C172 | 116.0(9) |
| O1-C171-C16 | 109.2(10) | O1-C172-C16 | 117.0(17) |

[a] Cent1, Cent2 are the centres of gravity of the cyclopentadienyl rings.

* Reference number with an asterisk indicates a note in the list of references.

larger metals (Sm) tridentate complexation is generally observed [12]. The structure clearly shows that the ether functions of both cyclopentadienyl ligands are coordinated to yttrium. The Y-O bond distances (2.438(5), 2.419(4) Å) are typical, and close to those found in complexes of other yttrium compounds with coordinated ether molecules $(MeC_5H_4)_2Y(THF)(\mu\text{-}H)_2$ (Y-O = 2.460(8)) [11]; $(Y(BH_4)_3(THF)_3$ (Y-O = 2.350(6), 2.412(7)) [13]; $[(1,3\text{-}Me_2C_5H_3)(THF)Y(\mu\text{-}H)]_2$ (Y-O = 2.46(1)) [14]. The $^1$H NMR spectrum of **3** shows a quartet (1 : 1 : 1 : 1) at $\delta$ = 0.62 ppm ($^1J$(B-H) = 83 Hz) for the terminal and bridging hydrides. $^1$H NMR spectra of **3** at various temperatures (cooling to -80°C in intervals of 10°C) did not reveal any splitting of the quartet into separate resonances for bridging and terminal hydrides, indicating a fast fluxional behaviour of the hydrides, as expected for this type of compound.

Compounds **2**, **3** and **4** show a strong $\nu$(C-O) absorption at 1044 cm$^{-1}$, shifted by 75 cm$^{-1}$ to lower wave number from the values for the stretching vibration of the free ethers [15*], so indicating a coordinated ether function. The $^1$H NMR resonances of the methoxy group are shifted to higher field for **2**, **3** and **4** compared with those for the free cyclopentadiene (3.09, 3.11 and 3.06 ppm, respectively; free cyclopentadiene: OMe = 3.36 ppm). Normally, coordination of the ether to a Lewis acid metal centre causes a shift to low field, as in $[(C_5H_4CH_2CH_2OMe)_2LaCl]_2$ [7] and $(C_5H_4CH_2CH_2OMe)_2TiCl_2$ [16]. Since the yttrium centre is a very strong Lewis acid and coordination of the ether function is obvious from the solid state structure we assume that this coordination is maintained in benzene solution. The reason for the high field shift remains unclear for the present.

In the $^1$H NMR spectrum of hydride **4** the hydride ligands give a triplet at $\delta$ 2.09 ppm ($^1J$(Y-H) = 28 Hz), indicating a symmetric yttrium hydride dimer, as has been observed for other systems such as $[(C_5H_4Me)_2Y(\mu\text{-}H)(THF)]_2$ ($^1J$(Y-H) = 27 Hz) [11], $[(C_5Me_5)_2Y(\mu\text{-}H)]_2$ ($^1J$(Y-H) = 38 Hz) [17*], $[(C_5Me_5)(OC_6H_3Bu^t_2)Y(\mu\text{-}H)]_2$ ($^1J$(Y-H) = 35 Hz) [18] and $[\{C_6H_5C(NSiMe_3)_2\}_2\text{-}Y(\mu\text{-}H)]_2$ ($^1J$(Y-H) = 28 Hz) [19]. Symmetrically bridged hydride ligands appear to be characteristic for yttrium and the lanthanides. Up to now only one asymmetric bridged hydride complex, $(C_5Me_5)_2Lu(H)\text{-}(\mu\text{-}H)Lu(C_5Me_5)_2$, has been reported [20].

## 3. Experimental section

All operations were performed under an inert atmosphere by use of Schlenk or glovebox techniques. Solvents (THF, ether, pentane, toluene) were distilled from Na/K alloy prior to use. $YCl_3(THF)_{3.5}$ was prepared from $YCl_3$ by continuous extraction with THF; $YCl_3$ was obtained by dissolving $Y_2O_3$ in hydrochloric acid and dehydrating the product with $SOCl_2$ [21]. IR spectra were recorded on a Mattson-4020 Galaxy FT-IR as Nujol mulls between KBr disks. NMR spectra were recorded on a Bruker WH-90, Varian Gemini 200 or Varian VXR 300. Chemical shifts are reported in ppm, $^1$H and $^{13}$C NMR spectra were referenced internally using the residual solvent resonances (benzene-$d_6$: 7.15 ppm, THF-$d_8$: 3.57 ppm, toluene-$d_8$: 7.09 ppm). Elemental analyses were carried out at the Microanalytical Department of this university. All values given are the average of at least two independent determinations.

### *3.1. $[(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}Cl)]_2$ (**1**)*

To a solution of 24.8 g (24 ml, 0.2 mol) of $C_5H_5CH_2CH_2OMe$ in 840 ml of toluene at 0°C were added, 30 ml of TMEDA and 78.5 ml (2.5 M, 0.2 mol) of n-BuLi, the mixture becoming orange. At -50°C 36.1 g (0.081 mol) of $YCl_3(THF)_{3.5}$ was added in portions and the mixture then allowed to warm to room temperature and stirred overnight (during the warming the mixture became bright yellow). Filtration, concentration, and subsequent cooling to -20°C yielded white crystals of **1** (19 g, 0.051 mol, 63%). $^1$H NMR (THF-$d_8$, 200 MHz): $\delta$ 2.65 (t, 4 H, CpC$H_2$); 3.52 (s, 6H, *Me*O); 3.91 (t, 4H, C$H_2$OMe); 5.85 (t, 4H, $C_5H_4$); 5.97 (t, 4H, $C_5H_4$). $^{13}$C NMR (THF-$d_8$, 75.429 MHz): $\delta$ 29.74 (t, Cp*C*H$_2$, $^1J$(C-H) 144 Hz); 79.40 (t, *C*H$_2$OMe, $^1J$(C-H) 142 Hz); 110.31 (d, *C*H ring, $^1J$(C-H) 165 Hz); 110.70 (d, *C*H ring, $^1J$(C-H) 165 Hz); 125.93 (s, *C* ring). IR (cm$^{-1}$): 3081m, 3023m, 2924vs, 2855vs, 1491m, 1460s, 1379m, 1335w, 1256w, 1229vw, 1202m, 1057vs, 1044vs, 993s, 966s, 918vw, 889vw, 833s, 816s, 781vs, 727w, 685m, 629w, 527m. Anal. found: C, 51.95; H, 5.92; Y, 23.92; Cl, 9.55. $C_{16}H_{22}O_2YCl$ calc.: C, 51.83; H, 5.98; Y, 23.98; Cl, 9.56%.

### *3.2. $(C_5H_4CH_2CH_2OMe)_2YAlH_4$ (**2**)*

To a solution of 1.53 g (4.1 mmol) of **1** in 50 ml of toluene at 0°C was added dropwise a solution of 2.4 ml (1.72 M, 4.1 mmol) of $LiAlH_4$ in THF. A white suspension was immediately formed. The suspension was allowed to warm to room temperature and stirred overnight. After filtration and concentration the solution was cooled to -20°C, affording 1.06 g (2.9 mmol) of **2** as white crystals (71 %). $^1$H NMR (benzene-$d_6$, 200 MHz): $\delta$ 2.27 (t, 4H, CpC$H_2$); 3.09 (s, 6H, *Me*O); 3.27 (t, 4H, MeOC$H_2$); 3.46 (s, 4H, Al$H_4$); 5.86 (t, 4H, $C_5H_4$); 5.98 (t, 4H, $C_5H_4$). $^{13}$C NMR (THF-$d_8$, 75.429 MHz): $\delta$ 29.45 (t, Cp*C*H$_2$, $^1J$(C-H) 127 Hz); 60.81 (q, *Me*O, $^1J$(C-H) 144 Hz); 78.16 (t, *C*H$_2$OMe, $^1J$(C-H) 145 Hz); 107.67 (d, *C*H ring, $^1J$(C-H) 163 Hz); 108.03 (d, *C*H ring, $^1J$(C-H) 163 Hz); 126.13 (s, *C* ring). IR

($cm^{-1}$): 3069w, 2955vs, 2924vs, 2855vs, 2728m, 2689m, 2674m, 2361m, 2342m, 1730s, 1456s, 1377m, 1200w, 1055m, 1042m, 993m, 966m, 920w, 895w, 835m, 797s, 775s, 741s, 681m, 523w. Anal. found: C, 52.35; H, 7.51; Al, 7.44; Y, 23.9. Calc.: C, 52.46; H, 7.16; Al, 7.37; Y, 24.27%.

### *3.3. $(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}H)_2BH_2$ (**3**)*

To a mixture of 0.85 g (2.2 mmol) of **1** and 0.29 g (13.3 mmol) of $LiBH_4$ at room temperature was added 40 ml of THF. The white suspension was stirred for 2 days and the solvent was then removed *in vacuo* and 50 ml of toluene added. White crystals of **3** were obtained, after filtration, concentration, and cooling of the toluene solution to -20°C (0.48 g, 1.4 mmol, 62%). Recrystallization from hot toluene gave crystals suitable for an X-ray structural determination. $^1$H NMR (benzene-$d_6$, 200 MHz): $\delta$ 0.62 (q, 4H, B$H_4$, $^1J$(B-H) = 83 Hz); 2.32 (t, 4H, CpC$H_2$); 3.11 (s, 6H, *Me*O); 3.29 (t, 4H, MeOC$H_2$); 5.90 (t, 4H, $C_5H_4$); 6.05 (t, 4H, $C_5H_4$). $^{13}$C NMR (toluene-$d_8$, 75.429 MHz): $\delta$ 28.99 (t, Cp*C*H$_2$, $^1J$(C-H) 128 Hz); 62.87 (q, *Me*O, $^1J$(C-H) 143 Hz); 78.67 (t, *C*H$_2$OMe, $^1J$(C-H) 143 Hz); 110.10 (d, *C*H ring, $^1J$(C-H) 164 Hz); 110.28 (d, *C*H ring, $^1J$(C-H) 165 Hz); 125.93 (s, *C* ring). IR ($cm^{-1}$): 3069w, 2953vs, 2924vs, 2855vs, 2726w, 2419w, 2367m, 2294w, 2226w, 2197w, 2174w, 1460vs, 1377s, 1304w, 1265vw, 1229vw, 1182vw, 1154vw, 1119w, 1055m, 1042m, 991w, 966w, 920vw, 837w, 812w, 785m, 762w, 723w, 681vw. Anal. found: C, 55.23; H, 7.50; Y, 24.93. $C_{16}H_{26}O_2YB$ calc.: C, 54.89; H, 7.49; Y, 25.39%.

### *3.4. $[(C_5H_4CH_2CH_2OMe)_2Y(\mu\text{-}H)]_2$ (**4**)*

To a solution of 0.64 g (1.7 mmol) of **2** in toluene/pentane (15/45 ml) was added 1.22 ml (8.7 mmol) of $Et_3N$, and the mixture was refluxed overnight during which metallic aluminium was formed. The solvent was removed *in vacuo* and the residue was extracted with toluene. Concentration of the extract and subsequent cooling to -20°C afforded **4** as white crystals (0.30 g, 0.9 mmol, 52%). $^1$H NMR (benzene-$d_6$, 200 MHz): $\delta$ 2.09 (t, H, $^1J$(Y-H) = 28 Hz); 2.56 (t, 4H, CpC$H_2$); 3.06 (s, 6H, *Me*O); 3.35 (t, 4H, MeOC$H_2$); 6.10 (t, 4H, $C_5H_4$); 6.22 (t, 4H, $C_5H_4$). $^{13}$C NMR (benzene-$d_6$, 75.429 MHz): $\delta$ 30.29 (t, Cp*C*H$_2$, $^1J$(C-H) 127 Hz); 60.73 (q, *Me*O, $^1J$(C-H) 142 Hz); 77.66 (t, *C*H$_2$OMe, $^1J$(C-H) 142 Hz); 107.25 (d, *C*H ring, $^1J$(C-H) 166 Hz); 108.06 (d, *C*H ring, $^1J$(C-H) 160 Hz); 123.87 (s, *C* ring). IR ($cm^{-1}$): 3069w, 2953vs, 2855vs, 2728vw, 1460s, 1377m, 1296m, 1262m, 1204w, 1184w, 1112m, 1061m, 1042m, 993vw, 968m, 881m, 866m, 829m, 812m, 768s, 752m, 704vw, 680vw, 635m, 615w, 526vw, 478vw. Anal. found: C, 57.26; H, 6.96; Y, 26.05. $C_{16}H_{23}YO_2$ calc.: C, 57.15; H, 6.89; Y, 26.44%.

### *3.5. X-ray structure determination*

Crystals of $C_{16}H_{26}O_2BY$ are orthorhombic, space group $Pna2_1$, with $a$ = 12.2448(7), $b$ = 11.1701(5), $c$ = 11.7887(5) Å, $Z$ = 4, $V$ = 1612.4(1) Å$^3$, MW = 350.10, $F(000)$ = 728, $\mu$(Mo K$\alpha$) = 36.4 $cm^{-1}$. X-ray data were collected at 100 K on an Enraf Nonius CAD4T/Rotating anode system [$\theta_{max}$ = 27.5°; $\omega/2\theta$-scan; $\Delta\omega = 0.53 + 0.35$ tg $\theta^0$; Mo K$\alpha$ monochromated $\lambda$ = 0.71073 Å] for a crystal [0.35 × 0.35 × 0.35 mm] mounted in a Lindemann glass capillary. Unit cell dimensions were derived from the SET4 setting angles of 24 reflections in the range $10 < \theta < 14°$. A total of 2161 reflections was scanned, corrected for Lp and absorption (DIFABS [22]) and averaged into a unique set of 1384 reflections with $I > 2.5\sigma(I)$. The structure was solved with DIRDIF-92 [23] and refined on $F$ to a final $R$ = 0.031, $R_w$ = 0.031 with SHELX-76 [24]. A disorder model was used to describe the positional disorder C171/C172. Hydrogen atoms were taken into account at calculated positions (except for those on B, for which the positional parameters were refined). Weights were based on counting statistics. The absolute structure was checked by refinement with opposite $\Delta f''$ contributions to the scattering factors. Scattering factors were those of Cromer and Mann [25], corrected for anomalous dispersion (Cromer and Liberman [26]). All calculations were done on a DEC5000/ULTRIX system. Geometrical calculations and the illustration were made with PLATON-92 [27].

TABLE 2. Final coordinates and equivalent isotropic thermal parameters of the non-hydrogen atoms for **3**

| Atom | $x$ | $y$ | $z$ | $U_{eq}$ (Å$^2$) |
|---|---|---|---|---|
| Y | 0.22194(3) | 0.05762(4) | 0.0092 | 0.0146(1) |
| O(1) | 0.1602(4) | 0.2362(4) | 0.1093(4) | 0.0290(16) |
| O(2) | 0.3507(3) | -0.1058(3) | -0.0064(4) | 0.0190(13) |
| C(11) | 0.2384(6) | 0.2580(7) | -0.1113(6) | 0.039(3) |
| C(12) | 0.1659(6) | 0.1837(8) | -0.1709(7) | 0.047(3) |
| C(13) | 0.2261(6) | 0.0872(7) | -0.2153(5) | 0.039(2) |
| C(14) | 0.3355(8) | 0.1014(8) | -0.1822(7) | 0.028(3) |
| C(15) | 0.3421(5) | 0.2061(5) | -0.1182(6) | 0.0257(19) |
| C(16) | 0.2093(11) | 0.3646(11) | -0.0450(12) | 0.076(5) |
| C(18) | 0.1224(7) | 0.2392(7) | 0.2244(6) | 0.040(2) |
| C(21) | 0.1310(4) | -0.1564(5) | 0.0305(6) | 0.019(2) |
| C(22) | 0.0933(9) | -0.0948(9) | 0.1254(7) | 0.026(3) |
| C(23) | 0.0215(8) | -0.0049(11) | 0.0881(8) | 0.029(3) |
| C(24) | 0.0158(9) | -0.0086(12) | -0.0300(8) | 0.030(3) |
| C(25) | 0.0830(9) | -0.1042(9) | -0.0649(6) | 0.025(3) |
| C(26) | 0.2134(4) | -0.2556(5) | 0.0281(8) | 0.023(2) |
| C(27) | 0.3261(5) | -0.2132(5) | 0.0592(6) | 0.0267(17) |
| C(28) | 0.4677(5) | -0.0890(6) | -0.0121(5) | 0.025(2) |
| C(171) * | 0.1444(13) | 0.3511(12) | 0.0662(12) | 0.029(3) |
| C(172) * | 0.2007(18) | 0.356(2) | 0.0620(19) | 0.029(3) |
| B(1) | 0.3606(9) | 0.0853(10) | 0.1981(9) | 0.028(3) |

$U_{eq}$ = 1/3 of the trace of the orthogonalized $U$. * Disordered.

Atom coordinates are given in Table 2. Tables of hydrogen atom coordinates and thermal parameters, and a full list of bond lengths and angles have been deposited with the Cambridge Crystallagraphic Data Centre. Lists of structure factors are available from A.L.S.

## Acknowledgments

Financial support by Shell Research B.V. is gratefully acknowledged. The investigation was supported by the Netherlands Foundation for Chemical Research (SON) with financial aid from the Netherlands Organization for Scientific Research (NWO).

## References and notes

1 P.L. Watson, *J. Am. Chem. Soc., 104* (1982) 337.
2 (a) H. Mauermann, P.N. Swepston and T.J. Marks, *Organometallics, 4* (1985) 200; (b) W.J. Evans, I. Bloom, W.E. Hunter and J.L. Atwood, *J. Am. Chem. Soc., 105* (1983) 1401; (c) G. Jeske, H. Lauke, H. Mauermann, H. Schumann and T.J. Marks, *J. Am. Chem. Soc., 107* (1985) 8111.
3 T. Sakakura, H.-J. Lauterschlager and M.J. Tanaka, *J. Chem. Soc., Chem. Commun.,* (1991) 40.
4 (a) M.R. Gagné and T.J. Marks, *J. Am. Chem. Soc., 111* (1989) 4108; (b) M.R. Gagné, C.L. Stern and T.J. Marks, *J. Am. Chem. Soc., 114* (1992) 275.
5 (a) K.H. den Haan and J.H. Teuben, *Recl. Trav. Chim., Pays Bas 103* (1984) 333; (b) K.H. den Haan, Y. Wielstra and J.H. Teuben, *Organometallics, 6* (1987) 2053; (c) G. Jeske, H. Lauke, H. Mauermann, P.N. Swepston, H. Schumann and T.J. Marks, *J. Am. Chem. Soc., 107* (1985) 8091.
6 (a) S.Ya. Knyazhansky, V.K. Belsky, B.M. Bulychev and G.L. Soloveichik, *Metalloorg. Khim., 2* (1989) 570; (b) Y.K. Gun'ko, B.M. Bulychev, G.L. Soloveichik and V.K. Belsky, *J. Organomet. Chem., 424* (1992) 289.
7 (a) C. Qian, D. Deng, G. Wu and P. Zheng, *J. Chem. Soc., Chem. Commun.,* (1990) 880; (b) D. Deng, F. Song, Z. Wang, C. Qian, G. Wu and P. Zheng, *Polyhedron, 11* (1992) 2883.
8 $AlH_3 \cdot NEt_3$ decomposes above 50°C with formation of metallic aluminium and $H_2$; *cf.* J.L. Atwood, F.R. Bennett, F.M. Elms, C. Jones, C.L. Raston and K.D. Robinson, *J. Am. Chem. Soc., 113* (1991) 8183.
9 After completion of the manuscript we became aware that **4** has been prepared independently from the chloride **1** by reaction with NaH; D. Deng, Y. Jiang and C. Qian, *Chin. Sci. Bull., 37* (1992) 348.
10 B.D. James, P.K. Nanda and M.G.H. Wallbridge, *Inorg. Chem., 6* (1967) 1979.
11 W.J. Evans, J.H. Meadows, A.L. Wayda, W.E. Hunter and J.L. Atwood, *J. Am. Chem. Soc., 104* (1982) 2008.
12 (a) M.F. Lappert, A. Singh, J.L. Atwood and W.E. Hunter, *J. Chem. Soc., Chem. Commun.,* (1983) 206; (b) T.J. Marks and G.W. Grynkewich, *Inorg. Chem., 6* (1976) 1302.
13 B.G. Segal and S.J. Lippard, *Inorg. Chem., 17* (1978) 844.
14 W.J. Evans, D.K. Drummond, T.P. Hanusa and R.J. Doedens, *Organometallics, 6* (1987) 2279.
15 (a) M. Hesse, H. Meier and B. Zeeh, *Spektroskopische Methoden in der organischen Chemie, 3 ed.*, Georg Thieme Verlag Stuttgart New York (1987), p. 50; (b) $\nu$(C-O) of the free ligand ($C_5H_5$-$CH_2CH_2OMe$) at 1119 $cm^{-1}$.
16 H. Qichen, Q. Yanlong, L. Guisheng and T. Youqi, *Transition Met. Chem., 15* (1990) 483.
17 Although initially reported as an asymmetric dimer (K.H. den Haan, Y. Wielstra and J.H. Teuben, *Organometallics, 6* (1987) 2053), detailed NMR studies on $[(C_5Me_5)_2Y(\mu\text{-}H)]_2$ in methylcyclohexane-$d_{14}$ have shown that the hydride ligands are symmetrically bridging; B.-J. Deelman and J.H. Teuben, manuscript in preparation.
18 C.J. Schaverien, J.H.G. Frijns, H.J. Heeres, J.R. van den Hende, J.H. Teuben and A.L. Spek, *J. Chem. Soc., Chem. Commun.,* (1991) 642.
19 R. Duchateau, C.T. van Wee, A. Meetsma and J.H. Teuben, *J. Am. Chem. Soc., 115* (1993) 4931.
20 P.L. Watson and D.C. Roe, *J. Am. Chem. Soc., 104* (1982) 6471.
21 J.H. Freeman and M.L. Smith, *J. Inorg. Nucl. Chem., 7* (1958) 224.
22 N. Walker and D. Stuart, *Acta Cryst., A39* (1983) 158.
23 P.T. Beuskens, DIRDIF-92, University of Nijmegen, Netherlands.
24 G.M. Sheldrick, SHELX-76, *Crystal structure analysis package*, University of Cambridge, UK, 1976.
25 D.T. Cromer and J.B. Mann, *Acta Cryst., A24* (1968) 321.
26 D.T. Cromer and D. Liberman, *J. Chem. Phys., 53* (1970) 1891.
27 A.L. Spek, *Acta Cryst., A46* (1990) C34.